

REMARKS

The Examiner has rejected claims 1-3 under 35 U.S.C. 103 over Johnson (US 5,520,708) in view of Kovacs (WO 03/040081). The Examiner takes the position that it would have been obvious for one skilled in the art to formulate the present invention upon a reading of Ergun. Applicants respectfully submit that this is not the case, since certain key features of the present invention are not taught or suggested by this reference.

The present invention relates to a method for improving the long term stability of biodiesels. The increased stability results from the presently claimed process is based on the elimination of crystallization nuclei by the action of the acid. This is achieved by a more intense washing, and a *hydrophilization* of the crystallization nuclei. Having recognized this mechanism for the flocculation in biodiesels and the possible counter actions, the present invention provides a method which is optimally suited to solve the problems known in the art. As described in detail below, the cited Johnson reference has only a single distribution between phases and utilizes a usual amount of an acid catalyst. This results in a biodiesels product prone to flocculation like other conventional biodiesels. The present application features a second distribution equilibrium and a higher acid content therein. The latter aids in the hydrophilization of the crystallization nuclei, which improves the extraction of the hydrophilized nuclei.

Johnson teaches a method for reducing the crystallization temperature of fatty acid oil ester-petroleum distillate fuel blends. Their process completely differs from the present invention in both materials and process steps. Johnson's process includes the combining of a petroleum distillate with an alcohol ester of a fatty acid, in an amount sufficient to lower the crystallization temperature of the blend. Johnson states that the esters may be prepared by conventional methods of transesterification of native oils with an alcohol such as methanol. Johnson further teaches the use of a basic catalyst which is mixed with the reactants. The reaction mixture is then extracted with hexane to induce a phase

separation. However, it is urged that this reference still fails to teach or suggest the specific process required by the present claims.

The instant invention relates to a method for improving the long term stability of biodiesels. Step (a) provides the forming of a reaction mixture comprising a crude methyl ester, by transesterification of a vegetable or animal fat or oil with methanol. Indeed such transesterification with methanol is known in general the art, and is mentioned in column 3 of Johnson. Step (b) of the present invention relates to a phase separation step, wherein a layer containing the crude methyl ester of step (a) is formed and separated from the rest of the reaction mixture. Such phase separation is also known in general in the art, and is disclosed via the addition of hexane and a water wash in Example 1 of Johnson. However, an important feature of the present invention, which is *not* taught or suggested by Johnson, is the present step (c) which requires an intensive inline mixing of the crude methyl ester layer obtained in step (b) at temperatures between 25 and 60°C with a strong acid or with a mixture of a strong acid and a complex former, to form an *emulsion*. Nowhere does Johnson provide such an intensive mixing step with a strong acid after their phase separation. In fact, nowhere does Johnson teach or suggest the formation of an emulsion *at all*. The word “emulsion” is not found anywhere in Johnson’s disclosure.

The Examiner agrees that Johnson fails to disclose – either explicitly or implicitly – that the methyl ester obtained after their phase separation is intensively mixed with a strong acid. However, the Examiner goes on to assert that Johnson does mention the use of a strong acid elsewhere in their disclosure. Applicants respectfully point out that Johnson only teaches the optional use of a strong acid as an acid catalyst in the transesterification reaction, as described in column 3 at lines 25-35. In fact, the reference goes on to state that such an acid catalyst is only used where large amounts of free fatty acids are present in the oil. Example 1 of Johnson clearly teaches that the oil and the alcohol are vigorously stirred together with sodium alcoholate. This is done to improve the reaction speed, because the oil and the alcohol do not mix together well (see Kovacs WO/03/040081 which states that it is known in the art that methanol and vegetable oil

have only limited miscibility in each other, and catalysts are used in order to accelerate the reaction). The main part of the acid, which according to Johnson is only present in amount of 0.1 to 0.5% based on the weight of the oil, is discarded with the separated lower aqueous phase. Applicants urge that it is neither mentioned in the text of Johnson, nor is it obvious in view of their disclosure, to use a strong acid *after* the transesterification reaction and *after* the phase separation of their process. It is submitted that the Examiner is impermissibly reconstructing the art in light of Applicant's disclosure, with no suggestion or motivation to do so.

The Examiner further agrees that Johnson fails to teach that the methyl ester resulting from step (b) is intensively mixed to form an *emulsion*. However, he cites Kovacs in an effort to fill the voids of Johnson, asserting that Kovacs teaches the use of high shear mixing in a homogenizer to form an emulsion. Applicants disagree, since first of all, Kovacs WO/03/040081 *incorrectly* states at page 3, lines 17-20, that U.S. 5,520,708 to Johnson teaches the use of a *homogenizer*. Applicants urge that no such homogenizer is taught in Johnson *at all*, and no mention of an emulsion is made anywhere in their disclosure. It is asserted that just because the reactants of their transesterification reaction are stirred vigorously *does not* indicate that an emulsion is formed. In fact, it is well known in the art that to form an emulsion with oil, water must be present. However, during the reaction phase of Johnson where their vigorous stirring takes place, no water is present. That is, water and hexane are added *after* the esterification reaction has been completed, in order to induce a phase separation. Thus, it is not possible for an emulsion to be formed according to Johnson's examples.

In addition, Applicants provide herewith a Declaration under 37 CFR 1.132 from inventor Dr. Rudolph Bönsch, stating that the formation of an emulsion of crude methyl ester and a strong acid as required by the present claim 1, step (c), is not possible in view of the teachings of Johnson. In his Declaration, Dr. Bönsch attests to the fact that it would be clear to one skilled in the art that to form an oil emulsion, a certain amount of water must be carried by the oil. Dr. Bönsch's Declaration further states that one skilled in the art would know that to form the presently claimed emulsion in step (c), the strong

acid must be incorporated in dilute aqueous form. The strong acid in the present invention is used for the hydrophilization of crystallization nuclei, that is, to make the nuclei wettable with water. Thus, it would be clear to those skilled in the art that the strong acid in this application must be incorporated in dilute aqueous form to result in an emulsion. In contrast, since Johnson adds their strong acid only as a *catalyst* in the transesterification reaction, the addition of an aqueous acid would not be desirable since it would hinder the esterification process. Johnson gives no information about the concentration of their acid catalyst. However, Dr. Bönsch's Declaration indicates that an expert in the art would know that it would be counterproductive to use acidic water in the implementation of the esterification process of Johnson since their acid is only used as a catalyst, and is used in those instances where a large percentage of free fatty acids exist in the oil. The acid would then have the task of catalyzing the esterifying of free fatty acids. Since the second product of this chemical reaction is water, the addition of an aqueous acid would hinder the esterification reaction according to the law of mass action. One skilled in the art would expect that the acids of Johnson must be anhydrous acids for their intended purpose in the reaction mixture. It should be noted that the issue of the present strong acid being in aqueous form has not addressed previously, since it was not in question regarding the previously cited prior art. Thus, Applicants feel the need to address this issue at the present time, since it has now become relevant in view of the Examiner's citing of Johnson.

Another important issue is that Johnson monitors their reaction by TLC and then *after* the completion of the esterification, adds a hexane/water mix to induce phase separation. Thereafter, the ester phase is washed with water. There is *no mention* of a *further* vigorous stirring of their mixture *after* the addition of the hexane/water mix or during the washing. The Examiner asserts on page 3 of the Office Action that based on Kovacs, it would be obvious for one skilled in the art to *repeat* the "vigorous stirring" step of Johnson with their ester-rich phase after the separation step. Applicants strongly disagree, since it is clear that Johnson's vigorous stirring occurs in their transesterification reaction process *prior* to phase separation only. Again, it is urged that *no repetition* of the vigorous stirring or the esterification step of Johnson is taught by the

reference. The Examiner asserts that a motivation to do so could be found in Kovacs. Applicants strongly disagree. While Kovacs relates to the transesterification of vegetable oils, they *do not* relate to a process for reducing the crystallization temperature of such fuel blends as taught by Johnson. Kovacs separates their oil into polar and apolar phases, followed by further refining of their apolar phase. Kovacs states at p.4 (WO/03/040081) that their *apolar* phase may be reacted again to obtain a desired *viscosity*. However, there is no teaching in Kovacs that a *polar* phase would need to be repeated. Furthermore, Kovacs teaches that such a repetition of their apolar phase is necessitated by the use of a homogenizer in forming an emulsion, which is not taught or used in Johnson.

For the reasons stated above, as well as in the Declaration, Applicants urge that no emulsion is taught or possibly formed by Johnson's process due to the fact that no water would be present in this step. Again, an aqueous strong acid must be used to form an emulsion according to the present step (c), while Johnson's esterification reaction would necessitate the use of an *anhydrous* acid catalyst if present at all. The Examiner goes on to state that a twice-reacted reaction mixture "can" be again extracted with hexane and washed with water to induce a second phase separation. Applicants urge that the Examiner is using an impermissible "obvious to try" standard of patentability.

Applicants further urge that there is a major difference in the makeup of the emulsions formed by the present invention, and the vigorously stirred mixture formed by Johnson. The present application forms an emulsion of acid in the separated ester-rich phase. In this phase there are only traces of the alcohol and by-products left, and it is emulgated with the strong acid. In contrast to this, Johnson has a completely different composition, no matter which time the Examiner assumes the mixture to be of equal quality. That is, if examining Johnson's reaction mixture before the addition of hexane/water, the mixture still contains all by-products and excess alcohol. If examined after the water washing point, the upper phase only has traces of acid left, since the acid was separated out with the lower aqueous phase. Thus, not only are the method steps of Johnson different from the present invention, but the methods result in the formation of completely different

mixtures. It is thus urged that the reactions taking place with acid, and the distribution behaviors are completely different as well.

Finally, step (d) of the present claims requires the separation of an ester layer from the emulsion of step (c), and then subjecting the ester layer to a thorough water wash and a subsequent drying. Again, since an emulsion is not formed by Johnson, either alone or in view of Kovacs, the present step (d) which relates to separating an ester layer from the *emulsion* cannot be deemed obvious in view of the cited art. Further, the Examiner agrees that Johnson does not teach a drying step. However, whether or not such a drying step is considered obvious on its own, it is submitted that the sequence of steps required by the present claims is not taught or suggested by Johnson and/or Kovacs, and would not have been obvious to one of ordinary skill upon a reading of the cited art.

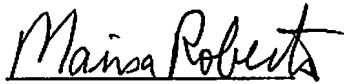
Regarding the Examiner's comments on claim 3, the Applicants wish to respectfully point out again that a separatory funnel is neither a wash column nor does it work with the counter current principle. A separatory funnel is *by definition* a single extraction stage which works batch-wise. Two immiscible liquids are introduced into the funnel, intensively mixed, and then allowed to separate again. There is no current at all.

For the above reasons, Applicants urge that it would not have been obvious to one of ordinary skill in the art to formulate the presently invention upon a reading of Johnson and/or Kovacs. It is therefore respectfully requested that the 35 U.S.C. 103 rejection be withdrawn.

The undersigned respectfully requests re-examination of this application and believes it is now in condition for allowance. Such action is requested. If the Examiner believes there

is any matter which prevents allowance of the present application, it is requested that the undersigned be contacted to arrange for an interview which may expedite prosecution.

Respectfully submitted,

A handwritten signature in cursive script that reads "Marisa Roberts". The signature is written in black ink and is positioned above the printed name.

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